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STATUS REPORT

EC-SR-11

ADVANCED FUEL CELL CATALYSTS

November 10, 1969 to February 10, 1970

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Research
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STATUS REPORT

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ADVANCED FUEL CELL CATALYSTS

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NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

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A. ABSTRACT, CONCLUSIONS, AND RECOMMENDATIONS

1. Abstract

As the main effort of this period has been concentrated on evaluation of catalyst performance, we are reporting details of electrode fabrication. Some of this work was performed, but not reported, during previous periods.

Life tests were instituted. One electrode has been tested for over 500 hours with no decay in characteristics.

The scanning electron microscope should be of advantage because of its high resolution and ability to display an entire small area. Several preliminary examples are shown in this report.

No adverse effects of glycerol traces in catalysts have been noted.

a. Comparative Measurements

The initial performances at standard testing conditions of various catalysts as mounted by us (with the exception of Ag black) in porous Teflon electrodes are shown in Table 1. Standard conditions are specified at the bottom of the table.

b. Life Tests

Nickel has not been available since the beginning of last August; therefore, our life-test gear has not been built as originally intended.

TABLE 1*
COMPARISON OF CATALYST PERFORMANCE (AVERAGE VALUES)

Catalyst	Volts at 100 mA/cm ²	mA/cm ² at 0.9 Volts
Pt black	0.800	40
AuCu + 22 at% Al (brown)	0.800	40
Ag black*	0.840	--
AuCu ₃ + 22 at% Al	0.875	70
Ag + 10 at% Al	0.890	85
AuCu + 22 at% Al	0.900	100
AuCu + 15 at% Al	0.910	115

Standard Testing Conditions:

O₂-H₂ pressures, 30 psi - 30 psi**

30-mil asbestos matrix containing 5 N KOH at 80°C

AB-40 anode

* private communication from Dr. Karl V. Kordesch

** Testing pressures of both gases have been raised from 15 psi to 30 psi, as formerly reported, to make use of greater forces aiding good electrical contact of electrodes and electrolyte.

One life test of electrodes is in operation, on a fuel cell loaned by Tyco Laboratories. While the performance of the electrodes appears satisfactory, the cell is not, as it shows nearly twice the internal resistance expected from our measurements with the same electrode in other cells, and from Tyco's results. The performance of the cell is lower because the electrodes have excessive internal resistance. Other reasons for this malfunction are not understood at present.

The cell has been disassembled twice in an effort to improve performance. During one period of operation lasting about a week, currents of 3.2 Amp (64 mA/cm^2) were recorded at voltages between 0.95 V and 0.96 V. Results at 3.2 Amp compare to some of the best obtained elsewhere, as reported in the literature available to us.

The cell has been in operation for 550 hours and is performing steadily. During this period no significant changes in electrode performance have been recorded.

c. Metallurgy

It now is possible to use an Attritor ball mill with alcohol rather than water. The amount of glycerol used in grinding has been reduced to 10%. As a result, smaller particle sizes appear possible. For example, a particle size of 0.3 micron has been attained.

Pseudomonas Ruginosus bacteria were used to remove glycerol traces from catalyst fines, in an experiment discussed in Section B.1. As a result, agglomeration was noticeably less than when the glycerol was not so treated. However, performance was not affected.

If free surface copper is present in the fines a blue coloration will appear in the solution. It is adequately removed by etching the finished electrode in 1 N HCl at 60°C for about 20 minutes, followed by a NH_4OH bath of similar duration. If necessary, the fines could be similarly treated.

2. Conclusions and Recommendations

The initial performance of all the fine crystal powders tested is noticeably superior to the performance of Pt and Ag black. The average particle size of the fine powders is 0.5 μ .

Particle-size reduction from an average between 2 μ and 3 μ to 0.5 μ (a reduction factor of 4 to 6) has increased the current density by a factor of 3. Performance at the larger particle size was about equal to that of Ag. The performance at the smaller size was 3 times that of Ag.

The performance of our electrodes is nearing 100 mA/cm^2 at 0.950 V (presently 67 mA/cm^2) and 200 mA/cm^2 at 0.900 V (presently 140 mA/cm^2) at standard conditions (Table 1). It appears that further size reduction (a size reduction by a factor of about 5 is contemplated) and the other steps discussed in the text (Section C.1) should insure these and higher performances.

It is recommended that this program be undertaken.

B. FACTUAL DATA

1. Metallurgy

During this report period, the work of the metallurgical section has been substantially routine catalyst preparation. However, the following changes were introduced:

1) The effective height of the shot tower was increased to approximately 9 feet from 5.5 feet. The new tower works satisfactorily; it allows greater temperature freedom in running the molten alloys through the crucible nozzles.

2) There has been concern about the possible effect of glycerol, used as a grinding medium, remaining as a surface film on the catalysts. This may possibly create trouble later, directly or perhaps, as a result of the breakdown of glycerol in KOH.

Dr. Robert Rutman, Professor of Biochemistry, University of Pennsylvania, suggested we try one of the several bacteria known to have a specific appetite for glycerol.

Five grams of AuCu were added to a culture of *Pseudomonas Rugginosus*. After 24 hours the bacteria were expected to have eaten all the glycerol believed present in the catalyst (a monofilm) and to have completed their life cycle.

By these methods, a 10^3 :1 reduction in volume of glycerol should be effected. The dead bacteria should break down in KOH, and the fractions should easily wash away. The glycerol-free catalyst was centrifuged and dried in a standard manner. The material compacted as usual but, unlike the untreated material, did not remain sticky and broke apart easily.

The catalyst so prepared performed normally as an electrode. However, no improvement in performance was noted.

3) Figures 1, 2, and 3 are three electromicrographs obtained by Professor Hay of Drexel Institute of Technology on a scanning electron microscope.

The first two are AuCu agglomerates: the first at 300 X, the second at 1000 X magnification. The third is a similar material amplified 3000 times. In the last one the single particles forming the agglomerate are beginning to be visible. The method is to be used to study particle-size distribution after break up of agglomerates. The micrographs are interesting to show even at this stage because of the splendid direct visual qualities of this type of electromicroscopy.

2. Electrode Preparation

The composition of the slurry that is spread on a gold-plated nickel mesh and cured into an electrode, is as follows: per cm^2 of electrode -- 40 mg finely divided ($< 0.5 \mu$) catalyst, 10 mg Fisher #38 graphite powder, 15 mg Baker analyzed reagent calcium carbonate powder, and 4.29 mg Teflon (from du Pont Teflon 30 suspension). For mixing all amounts are increased by 20%, because that is the average loss of material during mounting.

The catalyst agglomerates, which form during the final stage of preparation (separating the fines from a water suspension by centrifuging), are broken by manual grinding in a tungsten carbide mortar and pestle (Fisher Co.). Calcium carbonate is added during grinding. Since the catalyst powder is black and the CaCO_3 is white, the homogeneity of the mixture is judged by its color. When this mixture appears well interspersed, the graphite powder is blended into it. Sufficient water is added to form a smooth slurry.

CATALYST AGGLOMERATES

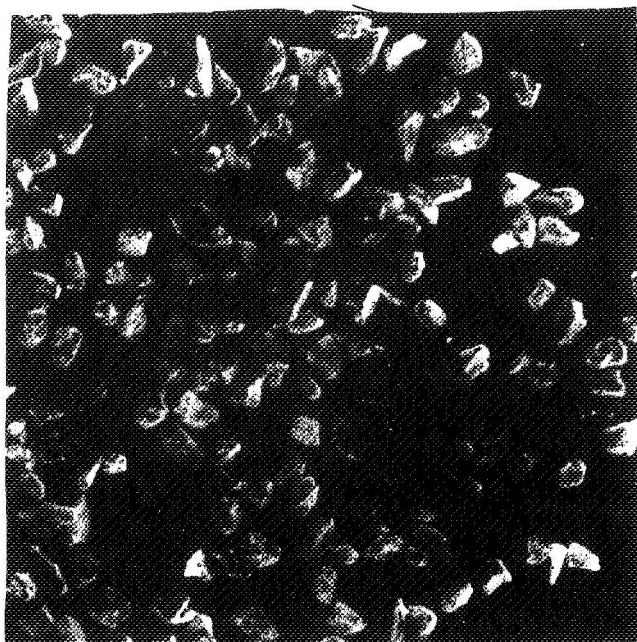


FIG. 1
300 X MAGNIFICATION

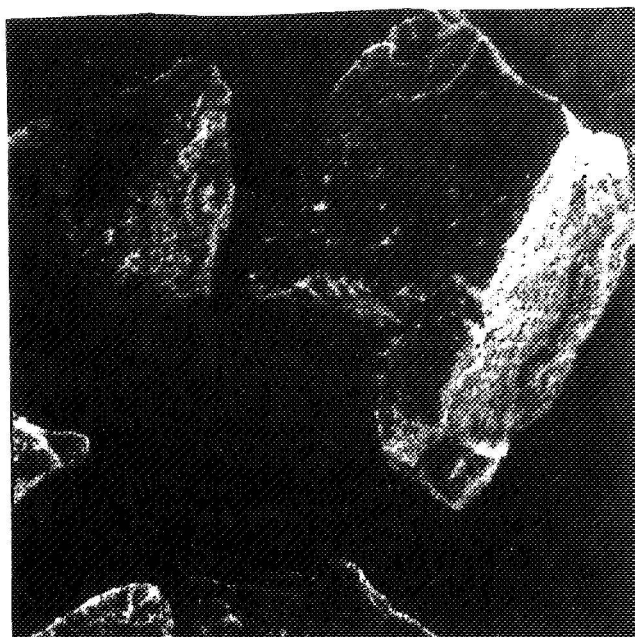


FIG. 2
1000 X MAGNIFICATION

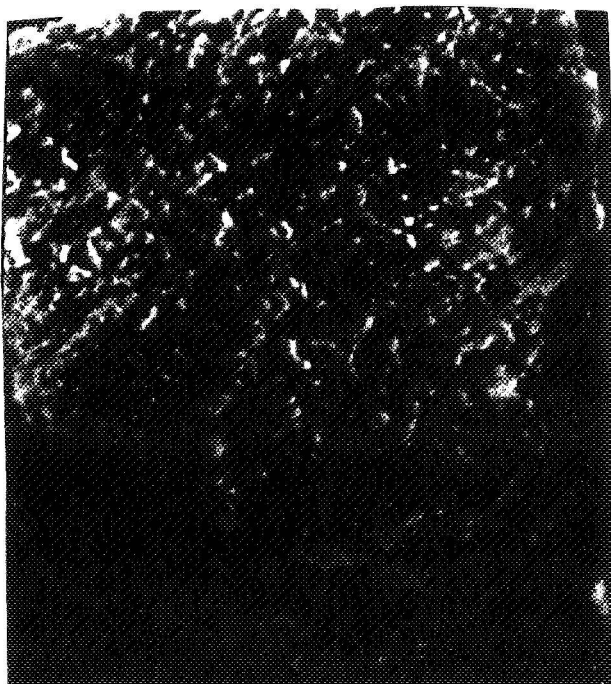


FIG. 3
3000 X MAGNIFICATION

0 1 μ 10 μ

Finally the Teflon is admixed. By blowing air over the slurry while continuously stirring, a smooth paste is formed which is then spread by spatula, onto the nickel mesh. This "raw" electrode is air-dried and pressed at 3000 psi in a Harco Lab press.

Electrodes are then sintered under argon (2 psig) in a Thermolyne hotplate oven set on a Chromalox hot plate. The temperature is raised to 300° in 20 minutes, held for 5 minutes, and lowered again to ambient in 10 minutes.

After sintering, the electrodes are etched in 1 N HCl at 60° for 20 minutes and washed in water, followed by a bath in 2 N NH_4OH for up to 20 minutes. This treatment is necessary to remove the CaCO_3 and such traces of surface copper as may be present.

Variations in this fabrication technique were performed to determine:

- 1) the amount of Teflon needed
- 2) the need for calcium carbonate
- 3) the need for pressure to form the electrode
- 4) the temperature of "curing"
- 5) the type of leaching solution and technique of rinsing

The first finely pulverized material was $\text{AuCu}_3 + 22 \text{ at\% Al}$ which had been ground in an Attritor ball mill containing glycerol. The glycerol was suspected of causing trouble in raising the particle contact resistance within the electrode. Although miscible with water, glycerol might form a tenacious monolayer which resists the repeated washings of the material after grinding. (See Section B.1, paragraph 2).)

Extensive testing through changes 1) through 5) above led us to our present standard 4.29 mg/cm^2 loading of Teflon, the electrode pressing technique, the curing temperature of 300°C , and the leaching process described below.

Both electrodes so produced appear to be adequate in comparison with nonproprietary information in the available literature but have much higher resistances than some electrodes now available.

The calcium carbonate is removed by etching in HCl, with the evolution of bubbles of CO_2 ; the rate can be easily observed. Some discoloration of the solution generally occurs. The well-known highly sensitive ammonium-hydroxide test will qualitatively indicate the presence of the copper ammonium ion complex. The presence of Cu ions is indicated by blue coloration. Our technique presently is to follow the etch with a rinse in a 2 N NH_4OH solution for about 20 minutes.

The conditioning effect of the etching treatments is shown in Fig. 4. This figure shows an early $\text{AuCu}_3 + 22 \text{ at\% Al}$ electrode which was made as described but apparently not adequately leached on the first trial. Poor performance led to a second etch. Testing showed improved performance and a further etch was attempted. Note the effect on the slope of the voltage-current curve in the range where cell resistance is an important factor. The upper curve was obtained after addition of approximately 50% more KOH than originally used.

One generally finds in the smaller short-term test cells that venting of hydrogen is effective in optimizing the voltage readout at a particular load. It is necessary to remove excess water that forms at the hydrogen electrode. In a simple rig, this is best achieved by bleeding H_2 . The H_2 is not recovered. However, this results in drying out of the electrolyte at the anode with a drop in performance at high current drain. Additional KOH is often needed to restore operation.

Fig. 4 : Effect of Acid Etching on Cathode Performance

Cathode: AuCu₃+ 22 at% Al 40 mg/cm²
 Teflon 4.3 mg/cm²
 Graphite 10 mg/cm²

Anode: AB-40

Curves are not IR-corrected.

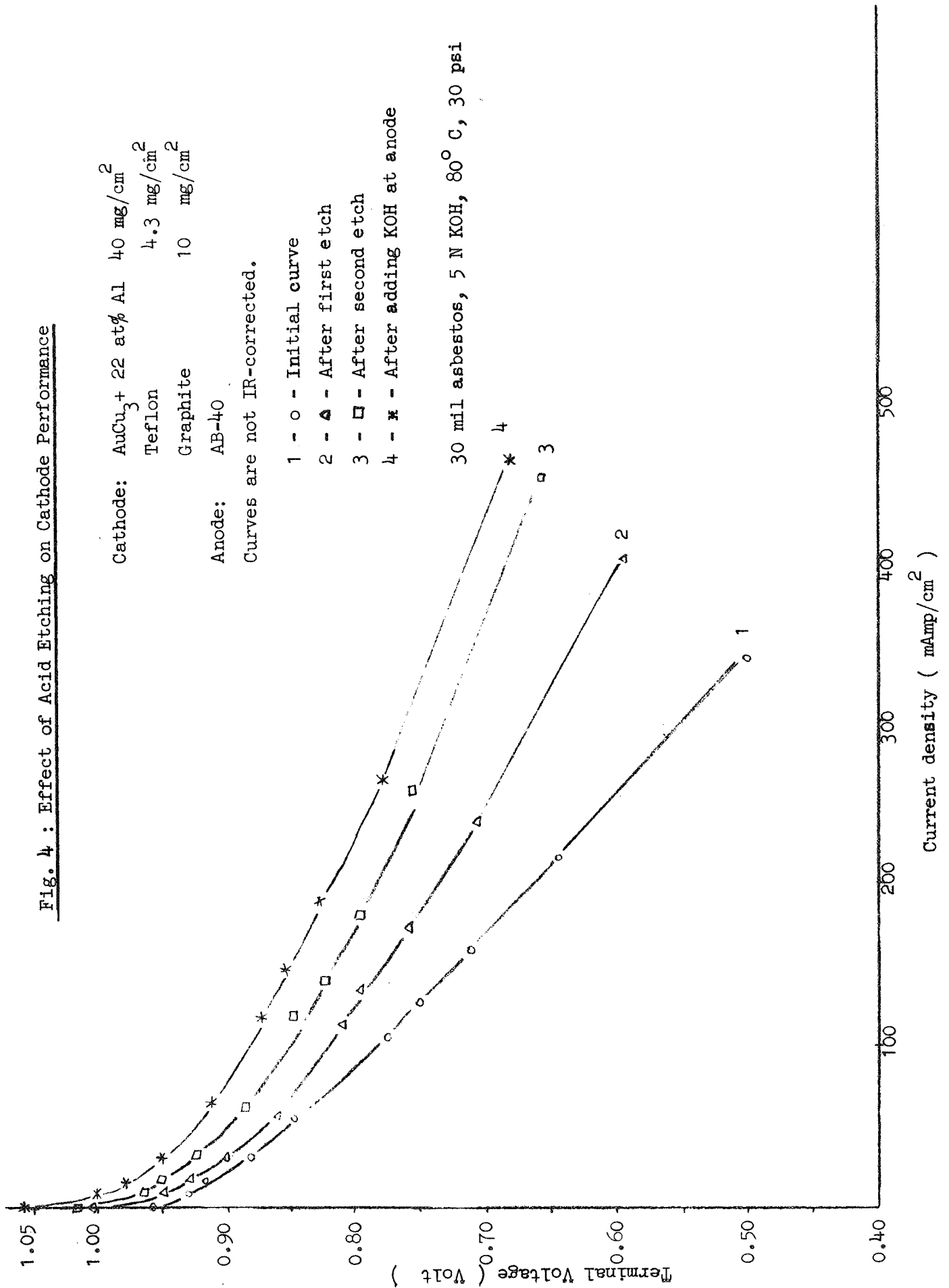
1 - o - Initial curve

2 - Δ - After first etch

3 - □ - After second etch

4 - * - After adding KOH at anode

30 mil asbestos, 5 N KOH, 80° C, 30 psi



Sometimes this additional KOH results in improved performance at every loading, as our figure indicates. The improvement can be explained as due to a decreased electrolyte resistance as the standard electrolyte normality is restored.*

Although the techniques of etching in H_2 and pressing at 3000 psi in a Harco Lab press were effective in producing good performance for $AuCu_3 + 22 \text{ at\% Al}$, when $AuCu + 15 \text{ at\% Al}$ was used we observed a considerable improvement in performance (Table 1). To date, three large electrodes of this material along with the nine small ones have been tested to show that the material performs well as an oxygen catalyst.

Etching is useful as a chemical conditioner of $AuCu + 15 \text{ at\% Al}$, as with other materials (see Fig. 6). Copper tends to leach out from this material, too, but less than from $AuCu + 22 \text{ at\% Al}$. The form of the lost copper is not known but presumed to be oxide, since free copper is relatively inert to HCl attack. Improperly alloyed copper in the crystal lattice may well form an oxide layer during comminution. Such an oxide layer could be deleterious, because copper oxide goes into solution in KOH (by comparison, free copper does not do so).

Pressing was investigated with $AuCu + 15 \text{ at\% Al}$, six small electrodes pressed and three not pressed. Average output voltage at 1 ohm load (about 60 mA/cm^2) was 930 mv for the pressed and 910 mv for the unpressed. The effect is even greater at high current densities (see Fig. 5).

* Reference curves of cell resistance vs KOH concentration: Fig. 5-8 and Fig. 5-9 of American Cyanamid Co. report NASA CR-54436, "High Performance Light-Weight Fuel Cell Electrodes" by R. G. Haldeman et al

Fig. 5: Effect of High Pressure in Electrode Fabrication

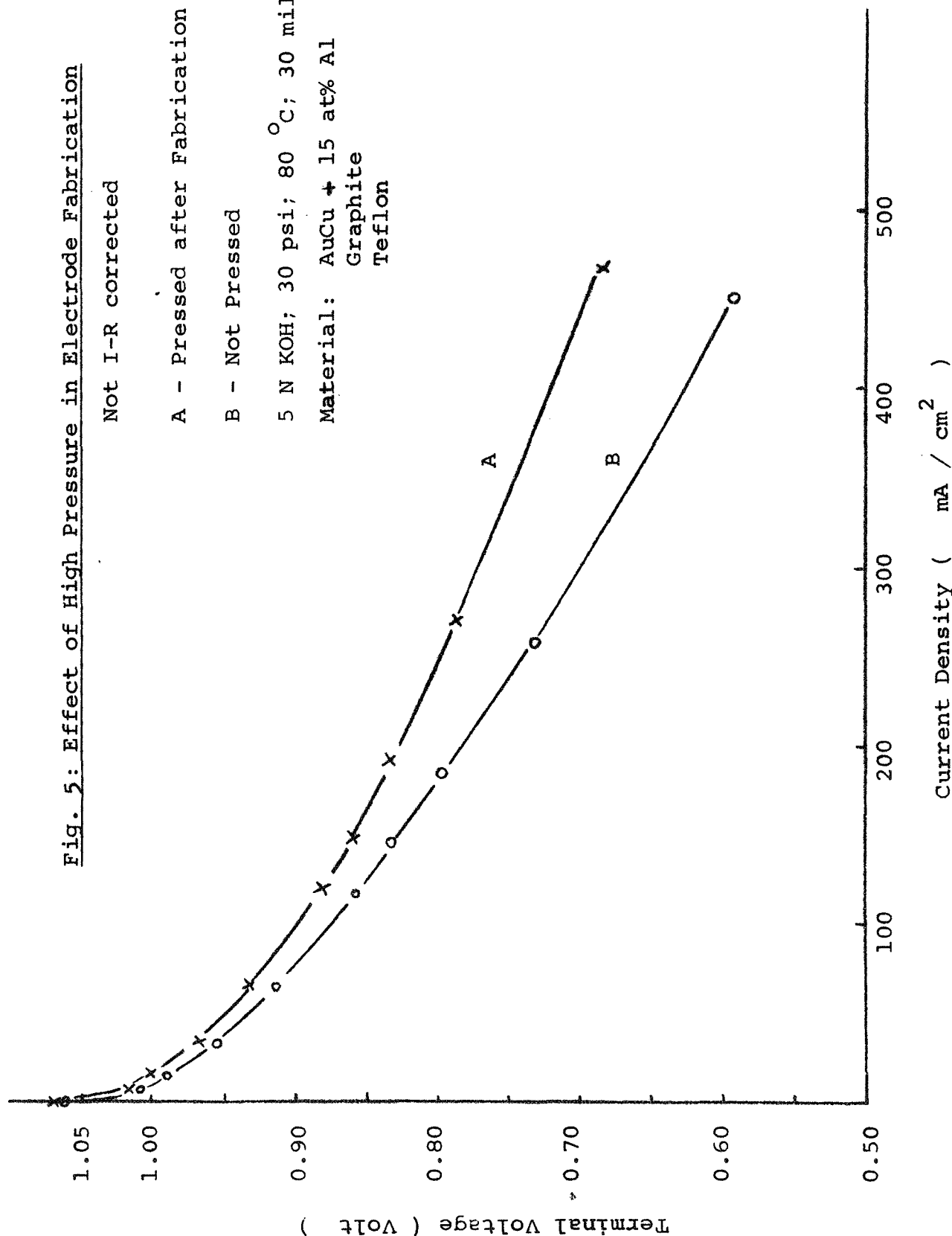
Not I-R corrected

A - Pressed after Fabrication at 3000 psi

B - Not Pressed

5 N KOH; 30 psi; 80 °C; 30 mil asbestos

Material: AuCu + 15 at% Al 40 mg/cm²
 Graphite 10 mg/cm²
 Teflon 4.3 mg/cm²

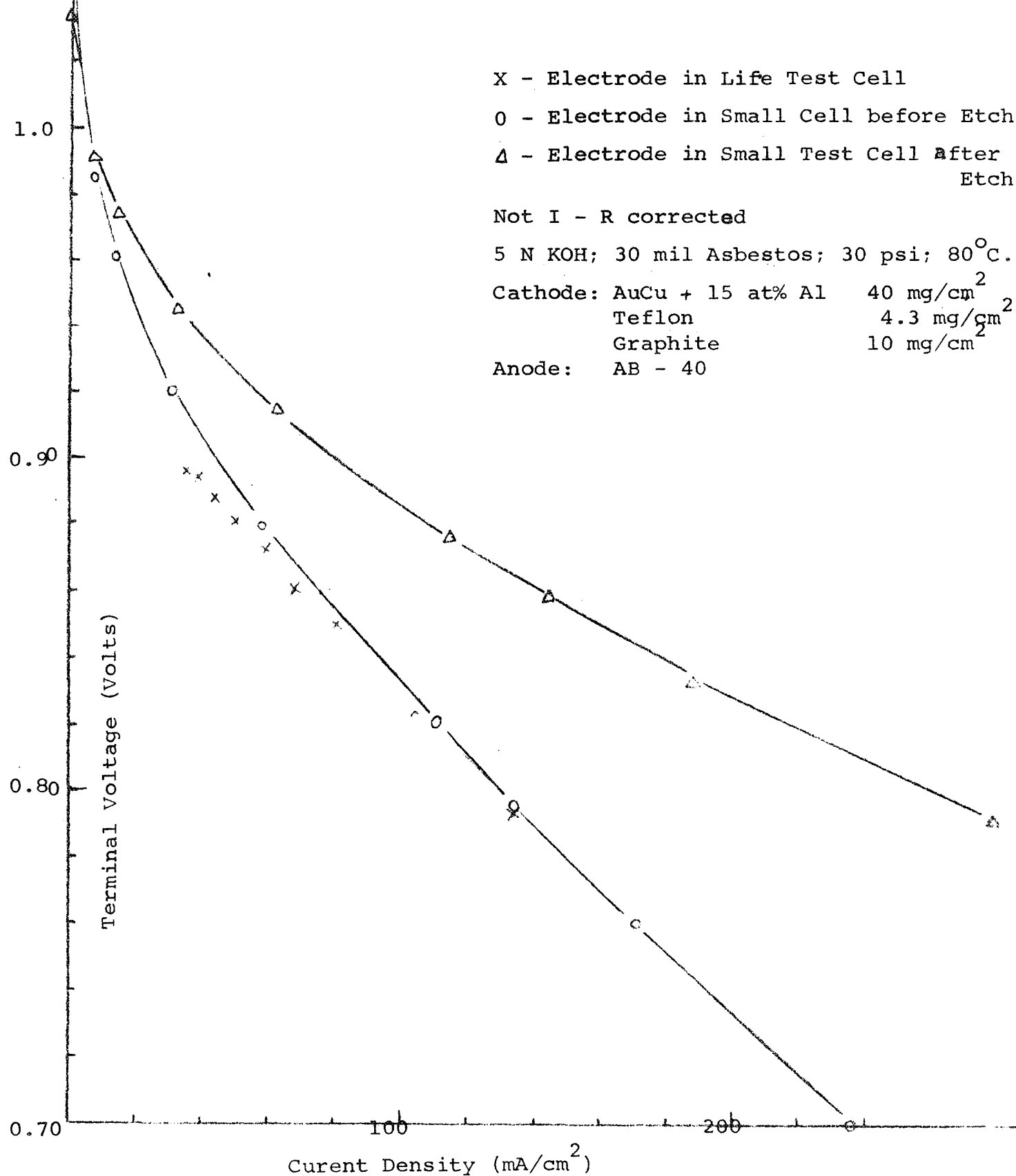


Two life-test cells have been operated to prove out this material. Only one has provided extensive continuous data; the other exhibits erratic variations with pressure which are difficult to identify. Therefore, our data for long-term operation refer only to one cell at this time.

One AuCu + 15 at% Al - 50 cm² electrode, B-7, showed poor life performance (equivalent 870 mv at 1 ohm). Smaller electrodes were cut from this electrode and the AB-40 and tested in a smaller cell. A 930 mv output at 1 ohm load was attained at the same operating conditions of 30/30 lb/in² O₂-H₂ pressure, 5 N KOH, 80°C; this is average performance for these electrodes. The difference in performance is probably a result of the fact that the cell was in fact constructed of stainless steel instead of nickel, and produced corrosion currents.

Fig. 6 shows the results obtained with electrode B-7. The zero-point scale is suppressed to show the effects more clearly. Initially we observed the same slope for the electrode in the small cell as in the large cell. Electrode etching, however, caused the slope to flatten out. The change in slope indicates reduced electrical resistance. Addition of KOH at the anode, as described in connection with Fig. 4, brought the performance to the 930 mv at 1-ohm load from the 915 value shown.

Further work remains to optimize performance. The chemical treatment needs to be standardized in order to suitably prepare a test electrode before installation and to avoid interruptions of performance.

Fig. 6: $H_2 - O_2$ Fuel Cell OperationComparison of Materials in Different Cells

3. Optimizing the Teflon Content

A series of electrodes were produced from $\text{AuCu}_3 + 22 \text{ at\% Al}$ ($< 0.5 \mu$ size) with Teflon content increased in small amounts from 3.73 mg/cm^2 of electrode to 4.675 mg/cm^2 of electrode as shown in Fig. 7. These amounts are nominal since actual amounts measured out are 20% larger to include the average loss of material during manufacturing.

Fig. 7 shows the performance as a function of Teflon content. A peak was established at approximately $4.44 \text{ mg Teflon/cm}^2$. The area around this apparent peak was then examined more closely, using $\text{AuCu} + 15 \text{ at\% Al}$ powder ($< 0.5 \mu$ size). Performance of this catalyst is generally superior to $\text{AuCu}_3 + 22 \text{ at\% Al}$, as was the case here. The relationship between terminal voltage and output current is essentially linear for low currents.

Electrodes were produced with 4.25, 4.29, and 4.39 mg Teflon/cm². The maximum activity was at 4.29 mg/cm^2 , which is equal to $5\text{-}1/18 \mu\text{l}$ Teflon 30 suspension per cm² of electrode. This is the amount used now.

Measuring such small amounts of liquid to the necessary accuracy is made possible by the R. Gilmont ultraprecision buret (capacity 2.5 ml) which reads amounts down to $0.1 \mu\text{l}$ directly.

4. Electrode Production

We have produced 360 electrodes of about 15 cm^2 area each. Twelve of these electrodes were made of commercial materials, such as Pt black, Ag, and Ni.

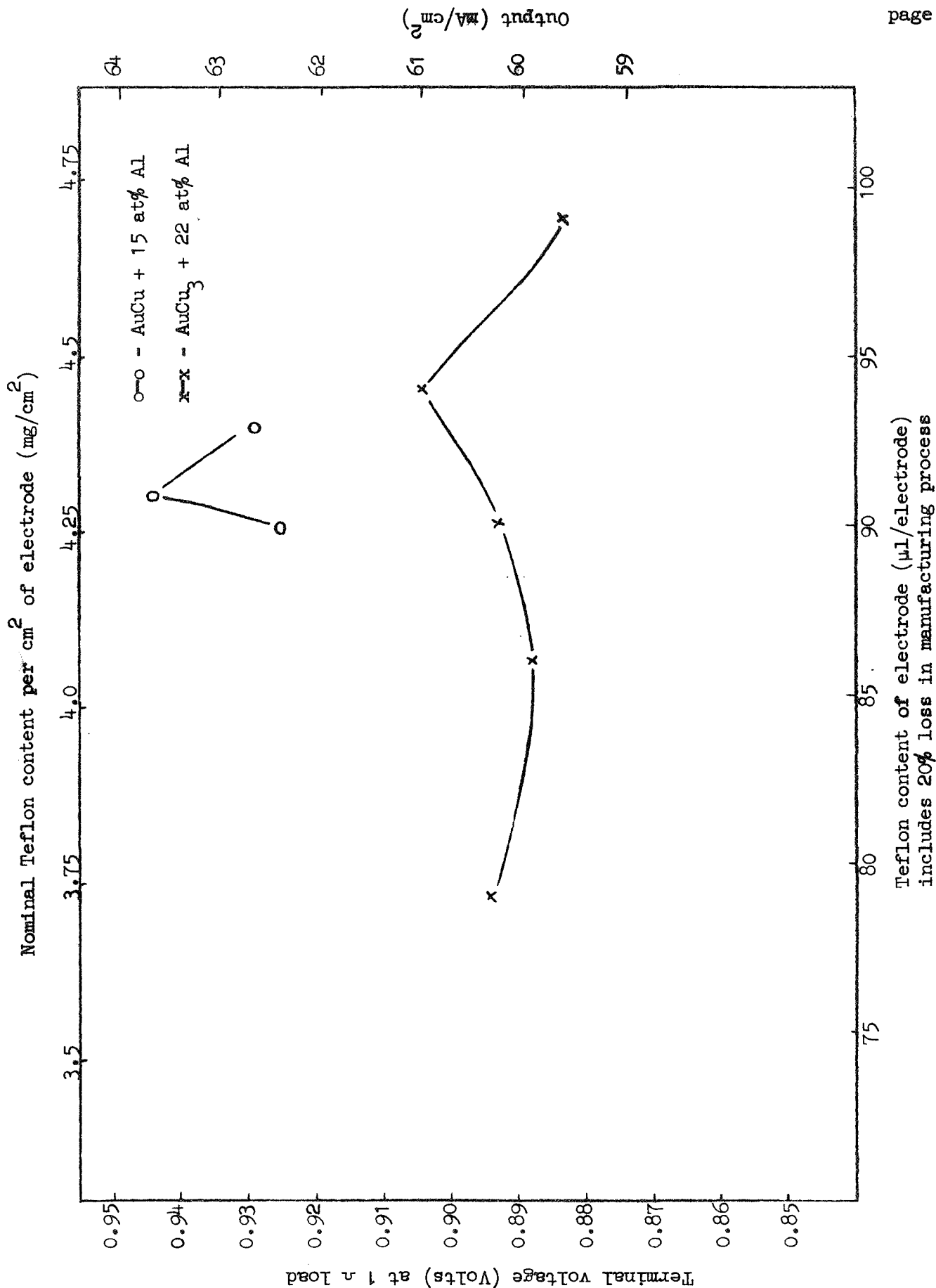


FIG. 7 -- PERFORMANCE AS A FUNCTION OF TEFLON CONTENT

Table 2 shows the number of electrodes produced with our own catalysts and tested to date.

TABLE 2
ELECTRODES

Basic Alloy Dopant		AuCu	AuCu ₃	Ag
0 at%				2
10 at%	Al	2	-	5
	In	-	-	-
	Ga	-	-	-
15 at%	Al	50	5	-
	In	-	9	-
	Ga	2	-	-
22 at%	Al	134	81	-
	In	33	-	-
	Ga	25	-	-
Totals		246	95	7

5. Catalyst Performance Measurements

The performance of catalysts under load has been recorded in small cells (14.8 cm^2) during short-term testing and larger cells (50 cm^2) during life testing. A number of techniques were tried to obtain better and more consistent results, as described below.

So far the best values have been obtained with a doped gold-copper alloy, AuCu + 15 at% Al, with AuCu + 22 at% Al a close competitor. In the small cell, nine different electrodes of finely ground blacks of the former material have been tested extensively and show a remarkable degree of similarity. Their spread does not exceed 20 mv for standard testing conditions (Table 1). Average results for this material are 100 mA/cm^2 current density at 0.9 volts and 250 mA/cm^2 at 0.80 volts under our standard conditions. Better than 550 hours of life testing in the 50 cm^2 cell have been accrued for this material with results which substantiate the small cell values.

a. Test Cells

We now describe our test cells in some detail.

1) The smaller cell is a nickel-plated brass unit consisting of two 4-inch diameter disks $3/4$ " thick, each of which has a milled cavity of $1-3/4$ " diameter, $5/32$ " deep, such that electrodes of a nominal area of 14.8 cm^2 are employed. Appropriate spacers consisting of gold-plated brass and nickel screen allow contact to be made to the electrodes. Three gas inlet and exhaust fittings with copper tubing ("gas in", "gas out", and "normally closed") are connected

through the two halves parallel to the disk axes. Asbestos matrices then are compressed between the two cell halves by holding screws, aided by the hydrogen and oxygen opposing pressures. The cell is mounted on a stand above a heater. Ordinarily, the oxygen side is on the bottom and the hydrogen side is on the top away from the direct heat.

Data are recorded with direct reading voltmeters (Honeywell Digitest Model 333 and United Systems Corp. Digitek D.C. voltmeter) and General Radio precision resistors in decade mounting of 0.1 and 1 ohm steps. Currents are deduced from voltage-resistance values and corresponding current densities from electrode area values.

2) The larger life-test cells are of Tyco design*. One modification of this cell, dictated by unavailability of nickel, was described in our Status Report August 10 - November 10, 1969.

3) An all-nickel cell has been loaned to us by Tyco Laboratories.

Essentially this larger cell is similar to the smaller cell with the following differences:

- a) larger electrode area of 50 cm^2 , solid nickel cell construction, including a solid nickel KOH reservoir of about 3 liter capacity,
- b) fine nickel mesh wicks dip into the reservoir to keep the lower electrode wet,

* For instance, J. Giner et al "Development of Cathodic Electrocatalysts for Use in Low Temperature H_2/O_2 Fuel Cells with an Alkaline Electrolyte", Contract No. NASW-1233 Q-12, Final Report, Jul 65 to Jun 68 by Tyco Laboratories, Waltham, Mass.

- c) hydrogen electrode mounted as the lower electrode which is wet by the wicks; water is removed by continuous flow of hydrogen,
- d) resistance load (required to obtain current densities corresponding to those measured in the smaller cell) is correspondingly lower, because current is more than 3 times that available from the smaller cell. Greater care of circuit assembly is required, with specially designed precision resistor assemblies.

b. Output Measurements

The dependence of fuel cell output measurements on circuit resistance values has been well described in the literature*. The slope of cell voltage versus current density can be considerably modified by small changes in attendant resistance. These resistances are the fixed cell resistance and the variable ones of the electrode-matrix assembly. The latter are associated with

- a) contact resistance between particles of the electrode,
- b) contact resistance at the current collector,
- c) resistance at the electrolyte-electrode interface, and
- d) electrolyte resistance, including that of the matrix.

* See, for instance, L. G. Austin, "Fuel Cells, A Review of Government-Sponsored Research 1950 - 1964", NASA SP-120, 1967, p. 44.

Recent effort has been to reduce particle size by extended Attritor ball-mill grinding. However, electrodes made of these fine particles did not perform as well as anticipated on the basis of correspondingly increased internal area. Very often these electrodes gave open circuit voltages several tens of millivolts too low, and the slope of the voltage current curves was much too steep. Indications were thus of either poorly fabricated electrodes and/or of catalyst material altered chemically or physically. We are investigating both possibilities.

6. Particle-Size Separation

Particle-size separation has received less time than scheduled during this period because of the following related facts:

- 1) improved performance of the $\text{AuCu} + 15 \text{ at\% Al}$ over the $\text{AuCu}_3 + 22 \text{ at\% Al}$, thus requiring attention to more extensive testing than planned,
- 2) poorer-than-expected performance of gas flotation for removing fines.

The separation assembly is completed and has been initially tested in the range of dry- N_2 flow rates anticipated, but results to date have not been encouraging. There is a tendency of the particles to agglomerate, and carryover by gas flotation appears to be hampered by other factors. Some fallout at the tube walls occurs. Extensive observations have not been carried out to determine the limiting factors.

In the present design, very little if any flotation of catalyst particles occurs -- perhaps because of a tendency of the particles to agglomerate. A tapping mechanism will be required. Because of the press of other work, work on the gas-flotation particles has been suspended.

7. Life Test Measurements

One electrode of 50 cm² area has been tested for over 500 hours continuously except for two short breaks. Quantitative results of this work are shown in Fig. 8 and 9.

This electrode was made to the specifications established by our small cell investigations of AuCu + 15 at% Al. Operation of the cell was monitored by a Varian G-22 A stripchart recorder. Values of voltage and resistance were measured as previously described with calibrations checked by means of an Eppley standard cell and a Wheatstone bridge.

Note in Fig. 8 the two breaks in operation were accompanied by relatively large changes in output. The first break after three days of operation showed a copper-like deposit on the asbestos matrix and AB-40. Both electrodes were leached in a solution of 1 N HCl and HNO₃ at 60°C for about 20 minutes and then rinsed in a 2 N NH₄OH solution for about 20 minutes. The blue copper ammonium ion appeared in the rinse of both electrodes.

After six additional days of operation the test was interrupted to inspect the electrodes for further evidence of copper and because the slope of the voltage-current curve was not satisfactory. At this time both electrodes were leached as before. Only traces of the copper ammonium ion were evident. No quantitative tests were made. Performance did not immediately return to previous values, but has increased steadily ever since, and now is back to peak values. The I-V slope did not improve materially.

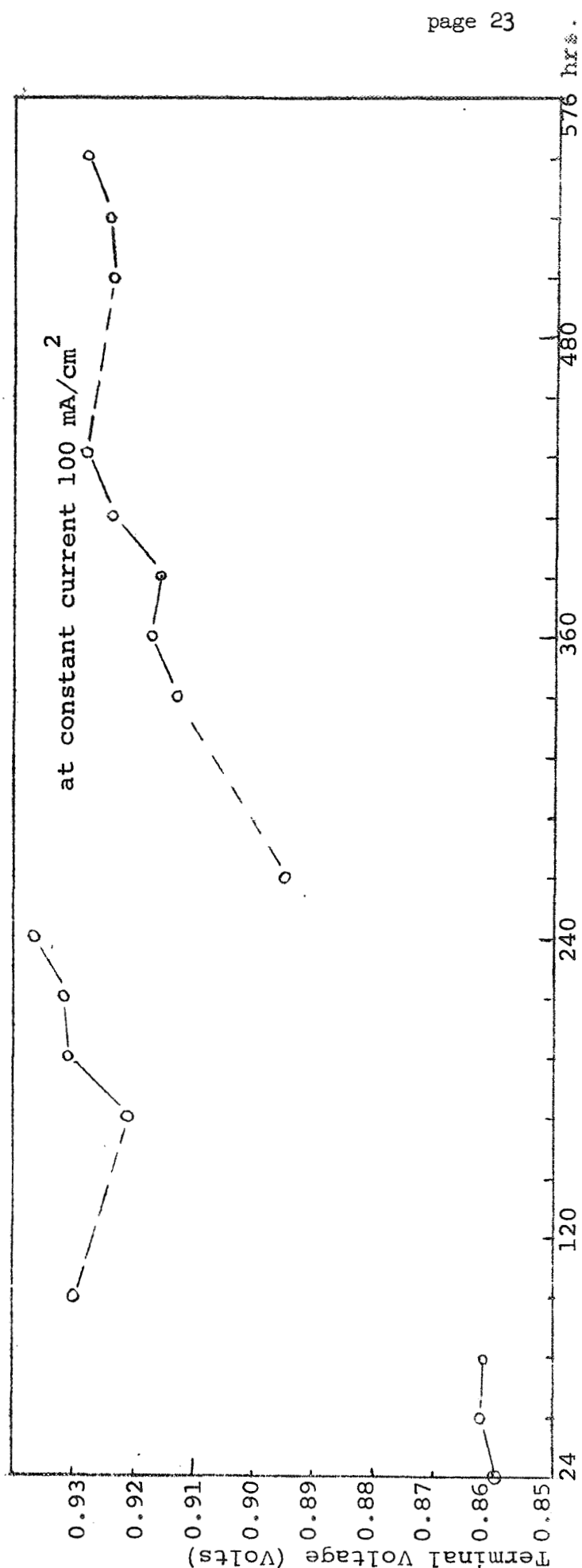
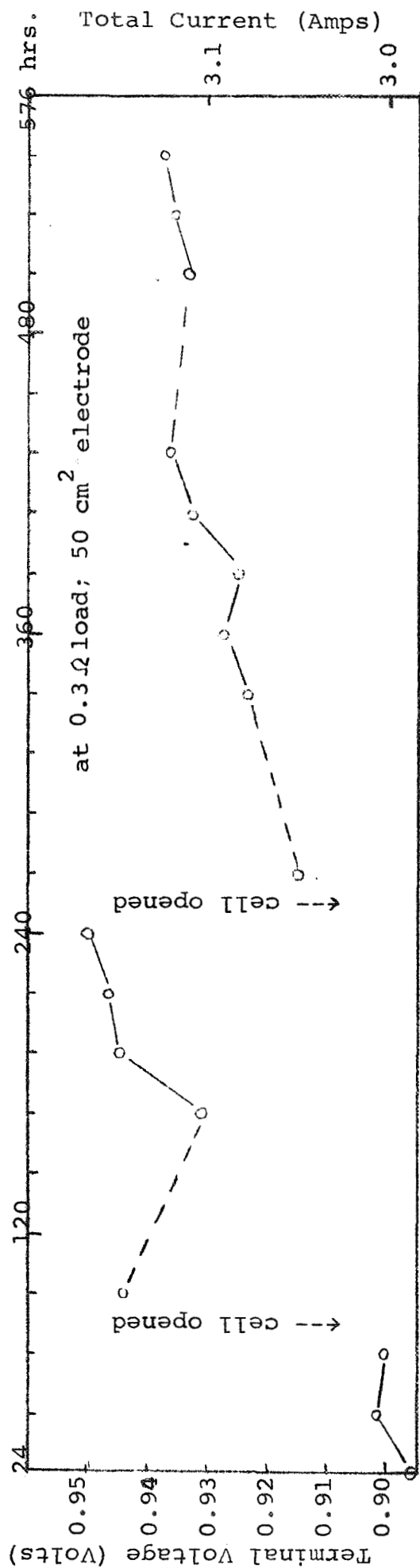


Fig. 8 : Life Test (Tyco cell, standard conditions).

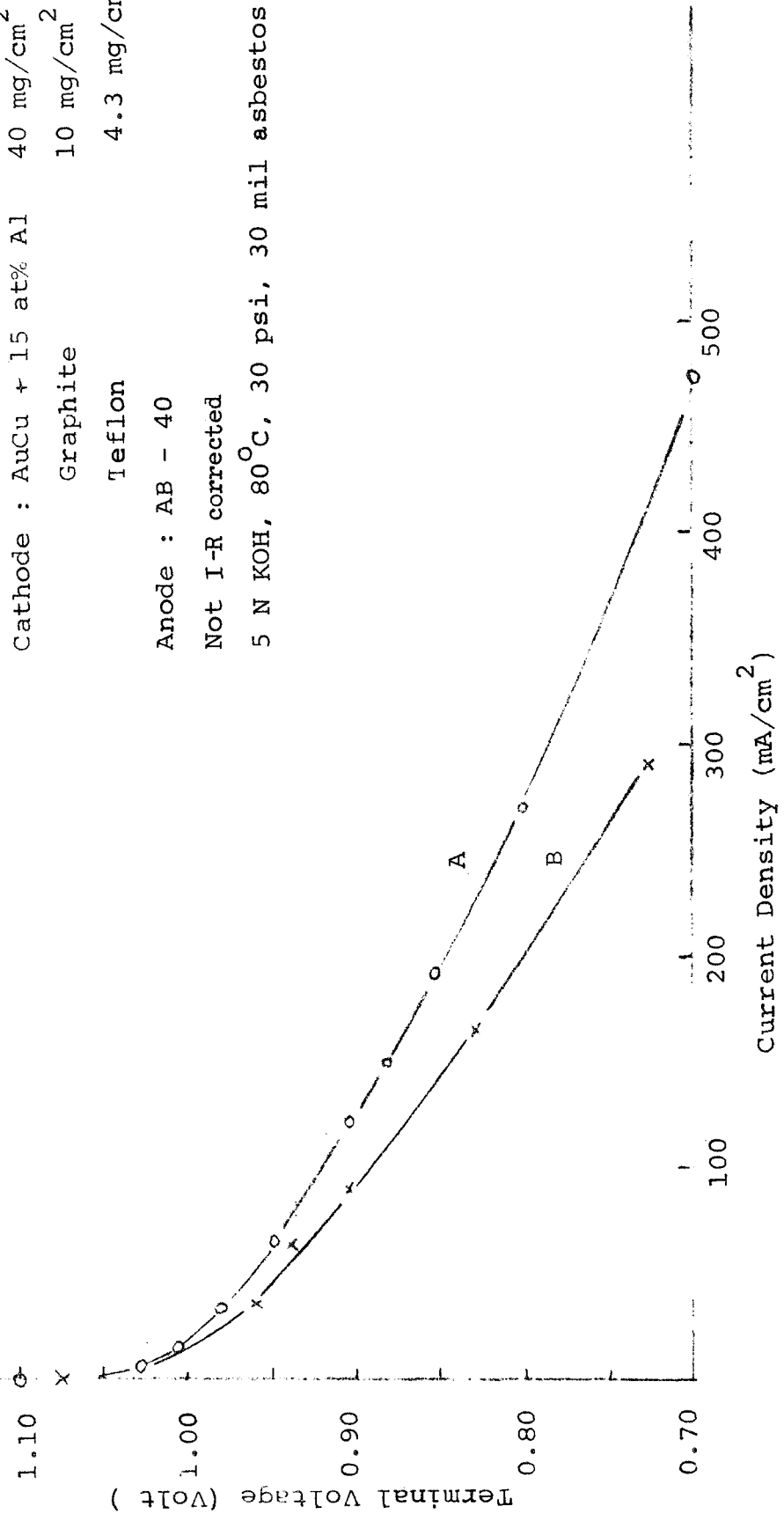
Fig. 9 shows a comparison of results from AuCu + 15 at% Al in both small and large cell operations. Note the difference in slope of these curves in the range of 100 - 300 mA/cm². Operation is similar at small current drains, but in the larger cell a larger internal resistance reduces the output considerably at higher current drains. Although voltage for a given load vary with life as Fig. 8 shows, and have improved by 20 - 25 mv for curve B, the slope after the first etch remains about the same. This suggests that the difference in slope is due to actual cell resistance rather than to the electrode material. The possibility of reducing the cell resistance will be fully investigated during the next period. The slopes of data presented by Tyco Laboratories for their life test cell show values that indicate this possibility, too.

Fig. 9 : Comparison of Short Term and Life Test Cells

A - Short Term (2hr) Operation, 14.8 cm² electrode
 B - Life Test (72 hr) Operation, 50 cm² electrode

Cathode : AuCu + 15 at% Al	40 mg/cm ²
Graphite	10 mg/cm ²
Teflon	4.3 mg/cm ²

Anode : AB - 40
 Not I-R corrected
 5 N KOH, 80°C, 30 psi, 30 mil asbestos



C. DISCUSSION

AuCu electrodes will be improved:

- a) by annealing the initial spherical pellets
- b) by etching the powders for surface Cu removal
- c) by improved grinding, as by substituting alcohol for water in the Attritor ball mill and by grinding under an inert gas
- d) by better separation of fines by air blast techniques, and possibly
- e) by further refining of electrode fabrication (i.e. rolling).

The performance of AuCu + 22 at% Al should be the same (or superior) to AuCu + 15 at% Al. AuCu + 22 at% Al will be tried again.

AuCu doped with Ga instead of Al gives similar values; In doping has produced lower performance. We plan to investigate the causes for these variations.

After AgCu was ground in the Attritor, it disintegrated in KOH. However, if ground in an aluminum-oxide mortar and pestle, it appears quite stable. It appears at present that the action of the Attritor promotes the oxidation of copper. We expect to repeat the experiment by running the Attritor under argon.

On the other hand Ag + 10 at% Al behaved well (cf. Table 1) and showed a distinct superiority to Ag black. This property of single crystal powders to outperform blacks had been expected. It should be verified with other materials, as most of the catalysts used can be produced in doped single-crystalline powders by our techniques.

D. PLANS FOR NEXT QUARTER

Just as the previous quarterly period concentrated on the production of adequate supplies of materials and the present quarterly period concentrated on testing and evaluating these materials, the final period will concentrate on life testing.